
पेट्रोलियम कोक — विशिष्टि

(दूसरा पुनरीक्षण)

Petroleum Coke — Specification
(Second Revision)

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum, and their Related Products of Synthesis or Biological Origin Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Petroleum coke is manufactured by delayed coking [Thermal Cracking] of residues obtained from crude oil distillation units, *primarily from vacuum distillation and sometimes from atmospheric distillation units*, of Petroleum Refineries. The petroleum coke thus obtained from different crude oil residues is categorised as Raw Petroleum Coke [RPC]. The petroleum coke, obtained by calcining the Raw Petroleum Coke [RPC] at a temperature of 1 200 to 1 400°C is known as Calcined Petroleum Coke [CPC].

The Raw Petroleum Coke [RPC] is either sold as such in the market or after calcination. RPC has limited application in industry, while the CPC is extensively used for the manufacture of carbon and graphite products and carbon anodes for the aluminium smelters.

India is one of the major producers and consumer of coal and coke on an extensive scale. National standards have been published, covering methods used for sampling and testing of coal and coke. But petroleum coke is comparatively a newcomer in the field of industrial applications and various industries, utilizing petroleum coke, have so far been guided by either ASTM or individual specifications and methods of test to suit their requirements. As a consequence of such practice, the major producers of this commodity are put to inconvenience. To overcome this difficulty, after collecting as much data as possible from various consumers and after giving due weightage to the various requirements, specifications and test methods, this Petroleum Coke Specification was first published in 1977.

This standard was subsequently revised in 1994. Requirements for volatile matter, sulphur and trace metals, that is, silicon, iron, vanadium and nickel for RPC have been modified.

The second revision of this standard is prepared after a thorough review of the standard in consultation with the major producers and consumers of these products. In this revision, requirements for volatile matter, sulphur and fixed carbon have been modified and specification requirements for premium grade of RPC have been incorporated. Test method for determination of moisture content by moisture balance is incorporated. A separate standard for petcoke used by aluminium industry is under preparation, keeping in view the sulphur content required in CPC for the equipment used by them.

Various documents relating to methods of test for coal and coke which are relevant and adaptable have been taken into consideration in this standard to avoid duplication of effort. The following Indian Standards for sampling and testing of coal and coke fall under the above category:

IS 436 (Part I/Sec 1) : 1964	Methods for sampling of coal and coke: Part 1 Sampling of coal, Section 1 Manual sampling (<i>first revision</i>)
IS 436 (Part 2) : 1965	Methods for sampling of coal and coke: Part 2 Sampling of coke, (<i>first revision</i>)
IS 1350 (Part 1) : 1984	Methods for testing of coal and coke: Part 1 Proximate analysis (<i>second revision</i>)
IS 1350 (Part 3) : 1969	Methods for testing of coal and coke: Part 3 Determination of sulphur (<i>first revision</i>)
IS 1354 : 1992	Methods for testing of coke: special tests (<i>second revision</i>)
IS 1355 : 1984	Methods of determination of chemical composition of ash in coal and coke
IS 7929 : 1975	Methods for determination of electrical resistivity of chemical coke

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PETROLEUM COKE — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for raw petroleum coke and calcined petroleum coke, used for the manufacture of electrodes, carbon and graphite products, carbon anodes and for other uses like manufacture of cement.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
IS 7929 : 1975	Methods of determination of electrical resistivity of chemical coke
IS 1447 (Part 4) : 1989	Methods of sampling of petroleum and its products: Part 4 Sampling of petroleum coke for laboratory analysis
1448	Methods of test for petroleum and its products
[P 33] : 1991	Part 33 Sulphur by bomb method
[P 79] : 1992	Part 79 Determination of trace elements in petroleum products — Vanadium
[P 126] : 1988	Part 126 Determination of ash content in raw and calcined petroleum coke
[P 127] : 1988	Part 127 Determination of iron in petroleum coke
[P 128] : 1988	Part 128 Determination of nickel in calcined petroleum coke
[P 130] : 1988	Part 130 Determination of vibrated bulk density of calcined petroleum coke
[P 131] : 1988	Part 131 Determination of silicone in petroleum coke

<i>IS No.</i>	<i>Title</i>
[P 132] : 1989	Part 132 Determination of moisture content in raw and calcined petroleum coke
[P 133] : 1989	Part 133 Determination of real density of calcined petroleum coke
[P 134] : 1989	Part 134 Determination of volatile matter in raw and calcined petroleum coke
[P 139] : 1992	Part 139 Determination of real density of calcined petroleum coke using butanol or toluene

3 TYPES

The material shall be of the following types and grades:

- a) Raw Petroleum Coke (RPC)
 - 1) *Premium* — Very low sulphur content
 - 2) *Grade A* — Low sulphur content
 - 3) *Grade B* — High sulphur content
- b) Calcined Petroleum Coke (CPC)
 - 1) *Grade A* — Low sulphur content
 - 2) *Grade B* — High sulphur content

4 REQUIREMENTS

4.1 The material shall be a petroleum product, free from all foreign matter and visible impurities.

4.2 The material shall also comply with the requirements given in Table 1, when tested according to the appropriate methods as given in col 7 and 8 of the table.

4.3 Optional Requirements

In addition to the requirements prescribed in Table 1, the material shall also comply with the requirements in **4.3.1** to **4.3.3** as agreed upon between the purchaser and the supplier.

4.3.1 *Size Analysis*

The product shall meet the requirements of size analysis, the limit and the method of test shall be as agreed upon between the *purchaser* and the *supplier*.

4.3.2 *Electrical Resistivity*

The product shall also meet the requirements of electrical resistivity; the values shall be reported as per two-electrode method of IS 7929, or by any other suitable method as agreed upon between the purchaser and the supplier.

4.3.3 *Trace Metals*

In addition to the requirements of trace metals, as given under Sl No. (viii) of Table 1, the material shall also meet the requirements of the following trace impurities. The limits and the methods of test for the determination of these trace impurities shall be as agreed upon between the purchaser and the supplier.

- a) Titanium (Ti); and
- b) Calcium (Ca).

5 SAMPLING

Representative samples of the material shall be drawn and prepared in accordance with IS 1447 (Part 4).

6 PACKING AND MARKING

6.1 *Packing*

The material shall be supplied in suitable containers as agreed upon between the purchaser and the supplier.

6.2 *Marking*

6.2.1 Each container shall be marked with the following information and any other information as agreed upon between the purchaser and the supplier:

- a) Name, type, grade and mass of the material;
- b) Indication of the source of manufacture, initials or trade-mark, if any; and
- c) Batch or code number.

6.2.2 *BIS Certification Marking*

Each container may also be marked with the Standard Mark.

6.2.2.1 The use of Standard Mark is governed by the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations made thereunder. The details of the conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

NOTE — In case of selling the material in bulk by road / rail / sea route, above clause 6, for Packing and Marking, shall not be applicable instead it will be decided as agreed upon between the purchaser and the supplier.

Table 1 Requirements for Petroleum Coke
(Clauses 3.2, 3.3 and 3.3.3)

Sl No.	Characteristics	Requirement for						Method of Test	
		Raw Petroleum Coke			Calcined Petroleum Coke		Annex	[P:] of IS 1448/ ASTM D	
		Premium	Grade A	Grade B	Grade A	Grade B			
(1)	(2)	(3)	(4)	(5)	(5)	(6)	(7)	(8)	
i)	Moisture content (as received) percent by mass, <i>Max</i>	10	10	10	—	—	A	Annex A / P : 132 / D 7582	
ii)	Moisture content (after initial drying), percent by mass, <i>Max</i>	2	2	2	0.1	0.1	—	P : 132 / D 7582	
iii)	Total Moisture content, percent by mass, <i>Max</i>	12	12	12	0.1	0.1		P : 132 / D 7582 / D 4239	
iv)	Ash content, percent by mass, <i>Max</i>	0.45	0.45	0.45	0.50	0.50	—	P : 126 / D 7582	
v)	Volatile matter,								
	a) percent by mass, <i>Min</i>	6	6	6	—	—	—	P : 134 / D 7582	
	b) percent by mass, <i>Max</i>	—	—	—	0.4	0.4	—		
vi)	Density:							P : 130	
	a) Vibrated bulk, g/cm ³	—	—	—	To be reported		—	P : 133	
	b) Real*, g/cm ³ , <i>Min</i>	—	—	—	2.03	2.03		and P:139	
vii)	Fixed carbon, percent by mass, <i>Min</i>	85	85	85	97	97	B	Annex B / D 7582	
viii)	Total Sulphur, percent by mass, <i>Max</i>	1.0	1.50	2.5	1.25	2.5	—	P : 33 / D 2622 / D 4239 / D 4294	
ix)	Trace metals:								
	a) Silicon (Si), percent by mass, <i>Max</i> .	To be reported		0.05	0.05	-	P : 131		
	b) Iron (Fe), percent by mass, <i>Max</i> .	-do-		0.04	0.04	-	P : 127 / D 3683		
	c) Vanadium*(V), percent by mass	-do-		0.03	0.03	-	P : 79 / D 3683		
	d) Nickel (Ni), percent by mass	-do-		To be reported		-	P : 128 / D 3683 / D6376		

(*) For graphite industry a higher real density and low vanadium content product is required; the limits for this may be settled between the purchaser and the supplier

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF MOISTURE CONTENT IN PETROLEUM COKE BY MOISTURE BALANCE

A-1 GENERAL

This test method covers determination of moisture (as received basis) in raw petroleum coke and Calcined Petroleum Coke (CPC) using moisture balance.

A-2 SUMMARY OF THE TEST METHOD

A known sample is placed in the moisture balance. The moisture balance records the weight of the sample. Determination of moisture content is initiated based on manufacturers operation manual. The sample is gradually heated to a predefined set temperature. At the set temperature, the sample is heated till weight loss in two consecutive readings is nearly constant. Loss in weight is recorded as moisture content in the sample.

A-3 APPARATUS

Any standard automated moisture balance for determination of moisture in solids.

A-4 PROCEDURE

The sample is crushed to ensure that does not have large lumps. The sample is homogenized by manual

mixing. About 20 gm. of sample is placed on the clean and dry pan of the moisture balance. The moisture balance will record the initial weight of the sample. Proceed with the determination of moisture content as per manufacturer's guideline to attain a set temperature of $107 \pm 3^\circ\text{C}$. During moisture content determination periodically instrument will measure the sample weight to determine the weight loss. Heating the sample and weighing the sample will be continued till instrument records constant weight loss in two consecutive determinations. When the weight loss is constant the instrument will display the moisture content on the screen.

A-5 REPORT

Report the moisture content up to single decimal point as percent, mass.

A-6 PRECISION

Precision may be calculated, as mentioned in the manufacturer's manual.

ANNEX B

[Table 1, Sl No. (vii)]

DETERMINATION OF FIXED CARBON IN PETROLEUM COKE

B-1 GENERAL

B-1.1 Fixed carbon in petroleum coke is the solid residue, other than ash, moisture and volatile matter, obtained by a process of calculation.

B-1.2 Total moisture, ash and volatile matter shall be determined by the methods given in IS 1448 [P : 132]; IS 1448 [P : 126] and IS 1448 [P : 134]

respectively.

NOTE — Fixed carbon is made up of carbon mainly, but may contain sulphur, hydrogen, nitrogen and oxygen as contaminants.

B-2 CALCULATION

Fixed carbon, percent by mass = $100 - (\text{Moisture} + \text{Ash} + \text{Volatile matter})$

ANNEX C

BIBLIOGRAPHICAL REFERENCES

<i>Other Standards</i>	<i>Title</i>
ASTM D 2622-16	Standard Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
ASATM D 3683-11	Standard Test Method for Trace Elements in Coal and Coke Ash by Atomic Absorption
ASTM D 4239-17	Standard Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion
ASTM D 4294-16	Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
ASTM D 6376-10	Standard Test Method for Determination of Trace Metals in Petroleum Coke by Wavelength Dispersive X-ray Fluorescence Spectroscopy
ASTM D7582-15	Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis

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Amendments Issued Since Publication

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